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## **EUROPEAN PATENT APPLICATION**

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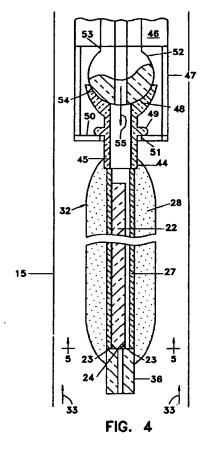
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## (54) Method of making optical fiber having depressed index core region

(57) Disclosed is a method of making an optical fiber preform having at least one annular region of depressed refractive index. A tube of silica doped with fluorine and/or boron is overclad with silica soot. A core rod is inserted into the overclad tube and the resultant assembly is heated while chlorine flows between the tube and the core rod to clean the adjacent surfaces. When the soot consolidates, the tube collapses onto and fuses to the rod. The resultant tubular structure is provided with cladding and drawn to form an optical fiber which exhibits low attenuation as a result of the low seed count at the interface between the inner core and the region that is doped with florine and/or boron.



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through the pores of a porous, cylindrically-shaped glass preform. The porous glass preform is heated to consolidate it into a non-porous fluorine-doped tube.

A boron-doped glass tube can be formed by depositing boron-containing glass particles on a cylindrical mandrel, removing the mandrel to form a tubular, boron-containing porous glass preform, and heating the porous glass preform to consolidate it into a non-porous boron-doped tube.

A further aspect of the invention concerns a method of making an optical fiber preform having an annular region containing a high content of fluorine. A tubular porous glass preform is initially formed. The preform is heated, and a centerline gas is flowed into the longitudinal aperture of the preform and outwardly through its pores. The centerline gas consists entirely of a fluorine-containing compound, whereby a high concentration of fluorine becomes incorporated in the pores of the preform. The porous preform is heated to consolidate it into a non-porous fluorine-containing glass tube. A cylindrically-shaped core rod is inserted into the fluorine-doped tube. The tube is then shrunk onto the core rod, and the interface between the core preform and the tube is fused.

#### **Brief Description of the Drawings**

Fig. 1 illustrates the formation of a porous glass preform on a mandrel.

Fig. 2 illustrates the consolidation of a porous glass preform.

Fig. 3 illustrates the application of a coating of glass particles to a fluorine-doped glass tube.

Fig. 4 as a cross-sectional view of an apparatus for consolidating and fusing the assembly formed by the method of Fig. 3.

Fig. 5 is a cross-sectional view taken along lines 5-5 of Fig. 4.

Fig. 6 is a cross-sectional view of the fused assembly resulting from the consolidation/fusion step illustrated in Fig. 4.

Figs. 7 and 8 are exemplary of the refractive index profiles of optical fibers that can be produced by the method of this invention.

Fig. 9 is a cross-sectional view of a draw furnace in which a tube is stretched and collapsed onto a rod.

#### Description of the Preferred Embodiment

The method of this invention produces an optical fiber preform having at least one annular region containing a refractive index decreasing dopant. Basically, this method comprises (a) making a glass tube containing a refractive index decreasing dopant throughout its entire radius, (b) inserting a core glass rod into the tube, (c) cleaning the adjacent surfaces of the rod and tube by flowing chlorine between them at an elevated temperature, (d) collapsing the tube onto the rod, and (e) adding to the resultant structure a sufficient amount of cladding that an optical fiber can be drawn therefrom. In one

embodiment, the tube is overclad with a soot coating. and steps (c) and (d) are performed in the same furnace, the overclad preform initially being subjected to an elevated temperature sufficient to achieve the chlorine cleaning, the temperature then being increased to consolidate the soot and collapse and fuse the tube to the rod. Fiber attenuation is low as a result of the low seed count at the interface between the inner core and the depressed index region resulting from step (c). The core of the resultant fiber includes the inner core region and the depressed index region and optionally includes other adjacent annular regions. In one embodiment of the invention, the annular preform region of depressed refractive index is doped with fluorine. Figs. 1 and 2 illustrate a method of making a fluorine-doped glass tube. Mandrel 10 is inserted through tubular handle 11. Mandrel 10 has a relatively large diameter in order to produce a tube having a sufficiently large inner diameter to be useful in later steps of the method. While mandrel 10 rotates, it also undergoes translational motion with respect to soot generating burner 13, whereby a porous glass preform 12 is built up on the mandrel.

A standard ball joint handle 14 (see handle 44 of Fig. 3 for greater detail) is fused to handle 11, and preform 12 is suspended in consolidation furnace 15 by that handle. Consolidation is performed in an atmosphere that includes a fluorine-containing centerline gas such as SiF<sub>4</sub>, CF<sub>4</sub>, C<sub>2</sub>F<sub>6</sub>, or the like. SiF<sub>4</sub> tends to give higher levels of fluorine doping (typically producing a -0.7% \Delta and occasionally producing a delta of about -0.8%), but that dopant causes elevated water levels in the resultant glass. Such elevated water levels in the fluorine-containing glass can be tolerated if the fiber core has a relatively high  $\Delta$ -value with respect to the silica cladding, whereby little power propagates in the annular fluorine-containing region of the fiber. CF<sub>4</sub> results in dryer glass but does not give the high dopant levels that can be obtained by using SiF4. High concentrations of fluorine can be used in this process because porous soot preform 12 is formed of pure silica, i.e. there is no dopant such as germania that could be disadvantageously diffused within the blank. The resultant consolidated tube contains a relatively high fluorine concentration since fluorine-containing gas is flowed into the tube aperture 18 (arrow 16) and outwardly through the pores of the porous glass preform whereby it achieves maximum contact with the entire body of porous glass, and since the centerline gas can consist of a pure gaseous fluorine compound that contains no diluent such as helium, chlorine or the like. Also, the only dopant introduced into the porous preform by the centerline flow is fluorine. The end of the porous preform that consolidates first preferably contains a capillary tube 19 to prevent the muffle gases from entering the preform aperture and to cause most of the centerline gas to flow outwardly through the preform interstices. A fluorine-containing gas also flows through furnace muffle 15, as indicated by arrows 17. Whereas the muffle gas 17 preferably contains a diluent gas such as helium and a sufficient amount of chlorine to dry the preform, the

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step would be undesirable for commercial purposes. Obviously, lower temperatures could be employed if processing time were not a concern. The flow of hot chlorine between the fluorine tube and rod 22 is very beneficial in that it allows the surfaces of the two members to be brought together without the formation of seeds at their interface. Seeds include defects such as bubbles and impurities that can produce attenuation in the resultant optical fiber.

As soot coating 28 consolidates, it exerts a force radially inwardly on tube 27, thereby forcing that tube inwardly against rod 22 to form a fused assembly 38 (see Fig. 6) in which the three regions 22, 27 and 28' are completely fused. A relatively low density soot provides a greater inwardly directed force; however, the soot coating must be sufficiently dense to prevent cracking.

Cladding glass layer 28' functions as part of the cladding in the resultant optical fiber. Fused assembly 38 is provided with additional cladding prior to drawing assembly 38 into fiber. For example, a coating of cladding soot can be deposited onto assembly 38 and then consolidated. Alternatively, assembly 38 can be inserted into a cladding glass tube.

In accordance with another aspect of the invention soot coating 28 is not deposited on tube 27, and tube 27 is not collapsed onto rod 22 in furnace 15. The assembly including rod 22, tube 27, tube 36 and ball joint handle 44 is subjected to an elevated temperature in a furnace while chlorine flows between rod 22 and tube 27 as discussed above. The temperature preferably remains within the range of about 1000°C to 1500°C to chemically clean the surfaces of members 22 and 27. After a sufficient period of time has elapsed to permit chemical cleaning to occur, the cleaned assembly 63 is removed from that furnace and is inserted into a conventional draw furnace (Fig. 9). The top end of rod 22 is provided with an enlarged end 65 which is suspended from a narrow region at or near handle 44. In the illustrated embodiment, the inside diameter of the bottom end of handle 44 is larger than the inside diameter of the top end of tube 27; this provides a ledge for supporting enlargement 65. A source of vacuum (not shown) is connected to handle 44. The bottom tip of assembly 63 is heated by resistance heater 62. As the tip of assembly 63 passes through heater 62, the diameter of the assembly decreases, and tube 27 collapses onto rod 22 and the space between those two members becomes evacuated. Further drawing of assembly 63 causes the assembly to elongate into a core preform rod 66 in which tube 27 is fused to rod 22. The core preform rod is severed into suitable lengths which are provided with cladding and drawn into optical fiber as described above.

Typical step-index optical fibers that were designed for use at wavelengths around 1300 nm exhibit a positive dispersion in the 1550 nm window where the fiber exhibits lowest attenuation. Such a system can be upgraded for operation in the 1550 nm window by placing in series with the step-index fiber a dispersion compensating (DC) fiber having a relatively high value of negative dispersion

at 1550 nm. The following example describes the manufacture of such a DC fiber.

A single-mode DC optical fiber having the refractive index profile illustrated in Fig. 7 was made as follows. A 0.25 inch (0.64 mm) alumina rod was inserted through the center an alumina tube having a 1.5 inch (3.8 cm) outside diameter. Rubber corks were used at the ends of the alumina tube to center the alumina rod within it. Handle 11 was placed near one end of the alumina tube. Pure silica soot was deposited on the alumina tube and on a portion of the handle. A detailed description of a method of forming a porous preform on an alumina tube can be found in U.S. patent 5,180,410.

A standard ball joint handle 14 was fused to the silica handle 11 prior to consolidation. Consolidation was carried out in the manner described in conjunction with Fig. 2. The centerflow gas 16 consisted of 1.5 slpm SiF4. Muffle gas 17 consisted of 20 slpm He, 0.5 slpm Cl<sub>2</sub> and 1.0 slpm SiF4.

The consolidated fluorine-doped tube contained about 2.4 wt. % fluorine (the ∆-value of the tube with respect to silica was about -0.7%Δ). The tube was redrawn to form an elongated tube having an outside diameter of approximately 12 mm and an inside diameter of 6.1 mm. A 30 inch (76 cm) long piece of fluorine-doped tubing 27 was severed from the consolidated tube. A standard ground joint handle 44 was fused to a first end of tube 27. A 4 inch (10 cm) long silica tube 36 having inside and outside diameters of about 3mm and 12 mm was fused to the second end of tube 27. The ends of the resultant tubular structure were mounted in a lathe where it was rotated and translated with respect to flame hydrolysis burner 13 (Fig. 3). Particles of SiO<sub>2</sub> soot entrained in the burner flame were deposited on tube 27 to build up a coating 28 having a length of 70 cm and a outside diameter of 90 mm. Coating 28 extended over the entire length of tube 27, and it extended a longitudinal distance of about 50 mm along handle 44. The coated structure 30 was then removed from the lathe.

The following method was used to make core rod 22. The large diameter end of an alumina mandrel was inserted into a glass tubular handle. The outside diameter of the mandrel tapered from 5.5 mm to 6.5 mm over its 107 cm length. The ends of the mandrel were mounted in a lathe where it was rotated and translated. GeO<sub>2</sub>-doped SiO<sub>2</sub> soot was deposited on the mandrel and a portion of the handle. The reactants GeCl<sub>4</sub> and SiCl<sub>4</sub> were initially flowed to the burner in sufficient quantities to form soot formed of SiO<sub>2</sub> doped with 37 wt. % GeO<sub>2</sub>. With each pass of the burner with respect to the mandrel, the flow of GeCl<sub>4</sub> was decreased, the last pass depositing pure silica soot. The flow of GeCl<sub>4</sub> to the burner decreased in accordance with such a recipe that the radial decrease in the concentration of

After the deposition of a soot preform to a thickness of 100 mm, the mandrel was removed by pulling it out through the handle, thereby leaving a longitudinal aperture. A capillary tube was inserted into the end of the

GeO<sub>2</sub> in the resultant fiber was substantially parabolic.

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- 4. The method of claim 1, 2 or 3 wherein said glass tube is formed by the steps of forming a tubular porous glass preform having a longitudinal aperture therethrough, flowing a fluorine-containing gas into said aperture and outwardly through the pores of said porous preform, and heat treating the porous glass preform to consolidate it into a non-porous fluorine-doped tube.
- 5. The method of claim 1, 2 or 3 wherein said glass tube is formed by the steps of depositing boron-containing glass particles on a cylindrical mandrel, removing the mandrel to form a tubular, boron-containing porous glass preform, and heat treating the porous glass preform to consolidate it into a non-porous boron-doped tube.
- 6. The method of 1 wherein, during the step of collapsing said tube onto said rod, said tube is evacuated.
- The method of claim 1, 2 or 3 wherein said chlorine containing gas consists of pure chlorine, or chlorine containing gas comprises chlorine and a diluent gas.
- 8. The method of claim 1 wherein the source of said centerline gas being continually supplied to the first end of said tube, and wherein the second end of said tube is subjected to a sufficiently high temperature that the second end of said tube collapses and prevents the further flow of said centerline gas, or a capillary tube being fused to the second end of said tube, the source of said centerline gas being continually supplied to the first end of said tube, and wherein said capillary tube is subjected to a sufficiently high temperature that said capillary tube collapses and prevents the further flow of said centerline gas.
- 9. The method of claim 1, 2 or 3 wherein said assembly is supported vertically during the steps of heating and flowing, whereby said centerline gas flows around the entire periphery of said core rod during the step of flowing.
- 10. The method of claim 1 wherein, during the step of collapsing said tube onto said rod, the step of flowing a centerline gas continues until it is discontinued by the collapsing of a softened glass member.
- 11. The method of claim 10 wherein an extension tube is fused to the second end of said tube, and during the step of collapsing said tube onto said rod, the step of flowing a centerline gas continues until it is discontinued by the collapsing of said extension tube.
- The method of claim 1 wherein the step of inserting comprises inserting a germania-doped silica rod into a fluorine-doped silica tube.

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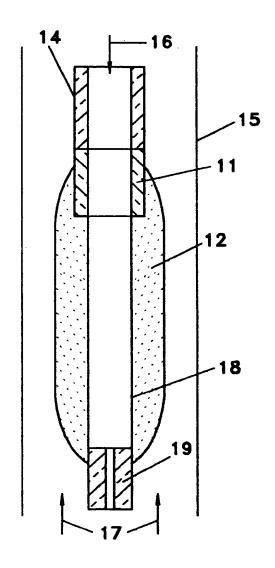
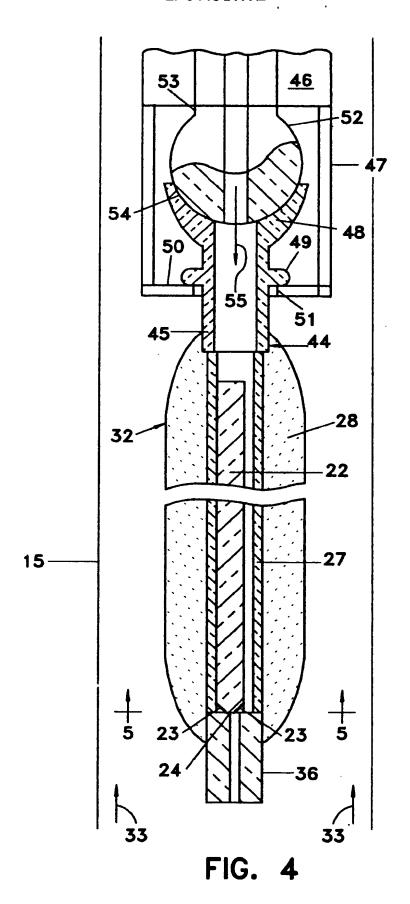


FIG. 2



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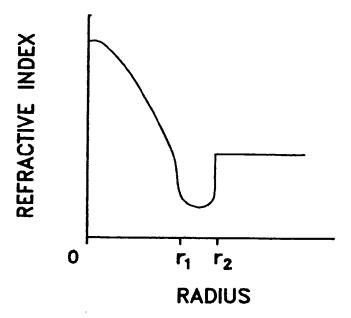


FIG. 7

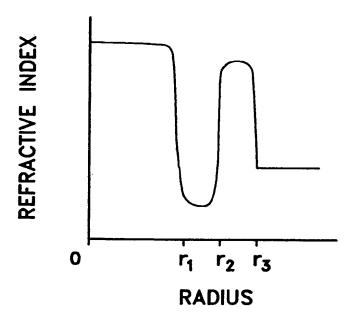


FIG. 8

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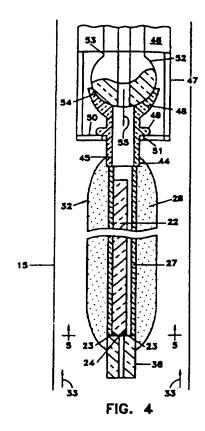
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- (71) Applicant: CORNING INCORPORATED Corning, N.Y. 14831 (US)
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# **EUROPEAN SEARCH REPORT**

Application Number EP 95 11 9160

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ategory	Citation of document with int of relevant pass		to claim	APPLICATION (Int.CL6)
A	EP 0 300 610 A (AT 8 * column 9, line 19 * claims 1-4; exampl	- line 46 *	1-12	
A	EP 0 359 351 A (CORM March 1990 * column 12, line 9 * claims; figures 1-	- line 17 *	1-12	
A	EP 0 147 225 A (CORM July 1985 * page 20, line 14 * claims; figures *		1-12	
A D	EP 0 182 250 A (SUM 1986 * page 4, line 17 - * claims; figures * & US 4 668 263 A	ITOMO ELEC IND) 28 Ma page 6, line 11 *	y   1	
D,A	US 5 180 410 A (BER January 1993 * column 3, line 62 * claims; figure 2	- column 4, line 42	1-12	TECHNICAL FIELDS SEARCHED (Int. Cl. 6)
D,A	US 4 486 212 A (BER December 1984 * the whole documen		1-12	
	The present search report has b	een drawn up for all claims		
	Place of search	Date of completion of the search	•	Examiner
	BERLIN	30 April 1997	Ku	ehne, H-C
Y:p:	CATEGORY OF CITED DOCUME articularly relevant if taken alone articularly relevant if combined with an ocument of the same category schoological background on-written disclosure	NTS T: theory or p  E: earlier pate after the fi  other D: document L: document	rinciple underlying the state of the same patent for other reason the same patent fan	on